

Solid Cryogen Formation in a Throttled Two-Phase Compressible Flow

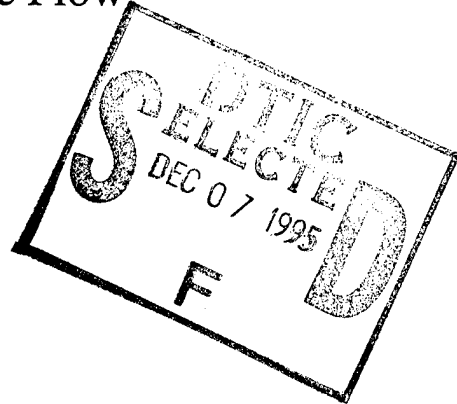
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Prepared by

R. L. WILLIAMS and R. P. WELLE
Mechanics and Materials Technology Center
Technology Operations

Prepared for

SPACE AND MISSILE SYSTEMS CENTER
AIR FORCE MATERIEL COMMAND
2430 E. El Segundo Boulevard
Los Angeles Air Force Base, CA 90245



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A handwritten signature in cursive script, reading "Pete A. Krawczyk", with a horizontal line extending to the right from the end of the signature.

P. K. Krawczyk, Capt. USAF
SMC/MEU

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Solid Cryogen Formation in a Throttled Two-Phase Compressible Flow

Randall L. Williams and Richard P. Welle †
Mechanics and Materials Technology Center
The Aerospace Corporation ‡
Los Angeles, CA 90009

Abstract

When the pressure of a liquid drops below its triple point, a phase transformation occurs forming both solid and gas phases with no remaining liquid. Liquid cryogens vented from launch vehicles, such as the Centaur Upper Stage, will undergo such a transformation when exposed to pressures below their triple points. Solids thus formed may accumulate in the overboard vent, restricting flow and possibly blocking the vent. An investigation of vent blockage was performed using saturated liquid argon. Observations of solid adherence and accumulation were recorded for various argon delivery pressures. Wall adherence and vent blockage was strongly dependent on delivery pressure, with accumulation increasing for decreasing pressure. For low delivery pressures, relative to the triple point, vent blockage was immediate and complete. In addition, solid formation was observed to propagate into the liquid, against the direction of flow, revealing the porous nature of the solid as well as the impact on the flow.

Introduction

For every liquid (except helium), there exists a pressure, called the triple point pressure, below which the material can no longer exist as a liquid. If the pressure of a liquid is reduced below the triple point pressure, the liquid must either vaporize or solidify. In an adiabatic depressurization, a portion of the liquid will vaporize with the remainder solidifying. The relative fractions in the solid and vapor phase will depend on the respective values of the heats of fusion and vaporization.

When the depressurization is a result of a flow from a high pressure region to a low pressure region, the phase transition will occur in the moving fluid. The solid thus formed may adhere to the walls of a flow channel. A buildup of solid on the walls can cause alteration of the flow path, resulting in a change of the pressure distribution and a translation in the location of the phase transition region. Under certain conditions,

buildup of the solid phase may close the channel and block the flow.^{1,2}

Because of a concern about possible solid formation during overboard venting of liquid hydrogen from the Centaur Upper Stage, a series of experiments has been undertaken in an attempt to understand the phenomena associated with solid formation in liquid cryogen flow systems.

Background

The Centaur is a liquid hydrogen/liquid oxygen upper stage rocket which has been in use with the Atlas and Titan launch vehicles since the 1960's. The liquid hydrogen is pressurized for injection into the combustion chamber of the Centaur's RL-10 engines by a two stage turbopump which is, in turn, driven by the expansion of hydrogen gas warmed by flowing through the combustion chamber and expansion nozzle cooling system. (The liquid oxygen is pressurized by a single stage pump which is driven through a gear train from the hydrogen pump.)

To prevent cavitation in the fuel pump, the two stage impeller section must be chilled to the boiling point of hydrogen (about 23 K at the hydrogen inlet pressure). Prior to launch, the fuel pump is chilled with cold gaseous helium from an external supply. Although pre-launch chill temperatures as low as 20 K have been used in the past, current practice calls for a pre-launch fuel pump temperature of about 100 K. This higher temperature was introduced after the Centaur failures on flights AC-70 and AC-71 were traced to condensation of solid air inside the fuel pump, preventing it from rotating. In any case, once the external helium supply is discontinued, the Centaur fuel pump begins to warm due to heat leakage from its surroundings. In normal operation, the Centaur engines are first fired some 250 to 300 seconds after launch. Before the engines can be started they must undergo what is known as the pre-ignition chill. The main fuel inlet valve is opened, allowing liquid hydrogen to flow through the fuel pump. During the pre-ignition chill, the hydrogen is vented through two overboard vent

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ducts. By this time, the vehicle is well above 20 km altitude where the background pressure falls below the triple point pressure of hydrogen (1.03 psia).

The flow system thus consists of a liquid hydrogen supply (at 23 K and 30 psia), a flow channel with some constrictions, and vents to a vacuum with an infinite pumping capacity. At some point in the flow path, the pressure must drop below the triple point pressure of hydrogen, leading to the formation and possible accumulation of solid hydrogen.

On the first Atlas-Centaur flight (AC-74) after the AC-71 failure, temperature data telemetry from the fuel pumps indicated that the pumps may not have cooled as rapidly as expected during the pre-ignition chill. A possible mechanism suggested to explain the slower than expected cooling rate was formation of solid hydrogen in the overboard vent ducts, leading to a partial or full blockage of the ducts and a reduction of the hydrogen flow rate through the system.

Although the observed anomalously slow cooling of the fuel pump was later traced to a problem with the temperature sensors, an experiment was initiated early on in the investigation to understand the mechanism of solid formation in a flowing cryogenic liquid.

Argon was chosen as a working fluid for the experiment for several reasons. The triple point pressure of argon is 9.97 psia, vs. ~1.03 psia for hydrogen. This allowed operation of the experiment at a substantially increased downstream pressure. The ratio between the heats of vaporization and fusion for argon is similar to that of hydrogen (5.51 vs. 7.79). A primary reason for using argon, of course, is the considerable relaxation of safety concerns. There are many additional materials properties which may affect solid formation and system plugging including density and viscosity of the liquid and gas phases, structural properties of the solid phase, ability of the liquid or solid to wet or adhere to the flow channel material, thermal conductivity of all three phases, and perhaps others. A list of properties for various fluids considered for experimental purposes is presented in Table 1.

A phase diagram for argon is illustrated in Figure 1. The phase diagram for hydrogen is similar in form, but with the boundaries occurring at differing temperatures and pressures. Each region displays the phase (gas, liquid, or solid) for a given temperature and pressure. Co-existence of two phases may only occur at region boundaries. All three phases may occur at one time for only one pressure/temperature combination known as the triple point, where all three regions intersect. For an adiabatic depressurization, such as that experienced in the overboard vent flow, liquid argon initially will cool through partial evaporation. The temperature and pressure will follow the liquid/gas

region boundary (saturation line). When the triple point is reached, a portion of the liquid will solidify while the remainder evaporates.

This work was intended to explore the significance of system pressures and flow geometries on solid accumulation and plug formation. Should further tests with liquid hydrogen prove necessary, this work will provide a framework for the layout of those tests.

Experimental Setup

Vaporization of a liquid cryogen in a partial vacuum can result in the production of large quantities of gas, leading to very large pumping capability requirements in a steady state experiment. Therefore, we elected to conduct the experiment in a "blow down" mode, using a 20 m³ vacuum facility as a dump tank for the expanding vapor. Solid formation in the flow would be possible as long as the tank pressure remained below the triple point pressure of the working fluid. In practice, the maximum available duration of the experiment ranged from 10's of seconds for high relative inlet pressure cases to essentially infinite for low inlet pressure cases with extensive plugging. In the latter case, the pumps normally used to evacuate the dump tank had adequate capacity (6500 torr-l/sec) to maintain a pressure below about 100 torr at the average flow rate through the intermittently plugged inlet.

To date, we have examined the basic phenomena of solid formation. This included studies of plug formation, plug propagation, blowout and plug cycling, phase transition location, wall adhesion, and plug porosity. The dependency of these phenomena on argon reservoir pressure and exhaust pressure was also explored. Delivery pressures of argon for the experiment ranged between 14.7 psia and 250 psia. In each case the argon was supplied as a saturated liquid.

The experimental apparatus constructed to provide a means of analyzing the phenomena was designed as a subscale, two dimensional simulation of the Centaur overboard vent duct. This required a high pressure manifold to feed a one inch square channel equipped with an interchangeable orifice. Figure 2 presents a schematic of the test section. Various orifice geometries were constructed to restrict the flow rate and vary flow characteristics if desired, but a thick-edged rectangular orifice designed to model flow from the Centaur overboard vent valve proved sufficiently enlightening. Following the throttle, a one inch square channel provided a 90° bend to the flow prior to exhausting into the evacuated dump tank.

The channel was constructed of black Lexan to minimize thermal loads on any solid formed as well as to

accentuate visual contrast with the white cryogenic solid. A clear Lexan plate was used to cover the channel and provided a transparent surface to observe the flow and solid formation. All tests were recorded on video tape. Pressure taps and thermocouples were instrumented at various positions in the channel to assist analysis.

Liquid argon fed the test section through a 1" ball valve which connected a delivery manifold to three ~200 liter liquid argon dewars. All were regulated to a maximum pressure of 250 psia. Lower pressures were achieved by venting the gas content within the dewars to atmosphere. The liquid delivery manifold, dewar feed lines and flow channel were pre-chilled with liquid nitrogen prior to testing. Tests were initiated by first opening dewar valves, then the 1" ball valve. The experiment was continued until the dump tank pressure exceeded the triple point pressure or until the experimental objective had been met.

Test Results

The results may be categorized into four groups. Each is referred to by the argon plenum (supply) pressure. *Extremely high*, *high*, *moderate*, and *slight* pressure ratings refer to the pressure ratio between the plenum and the triple point; $6.5 < \text{extremely high}$, $5 < \text{high} < 6.5$, $3 < \text{moderate} < 5$, $\text{slight} < 3$. Ratings are categorized by the nature of solid formation and adherence to the flow channel. Typical solid accumulation and adherence is graphically presented in Figure 3. These ratings are strongly dependent on the specific fluid and flow geometry, but phase transition similarities may be expected for other fluids experiencing similar flows.

Initial tests were conducted with the argon reservoir in the *extremely high range* at approximately 250 psia (ref. Figure 3a). Although solid was formed downstream of the throttle, no solid was observed to adhere to the wall in any stable fashion. High momentum in the flow appeared to prevent solids from accumulating and adhering to channel walls. Therefore, pressures were decreased to facilitate solid accumulation in the channel. A range of pressures between 65 to 30 psia in the argon reservoir produced solid which adhered to channel walls.

Pressures between 65 to 50 psia, *high range*, deposited significant solid in the channel downstream of the throttle (ref. Figure 3b). Immediately downstream of the orifice exit, solid was formed in the exit wake. This quickly accumulated in the recirculating regions downstream of the throttle on both sides of the orifice. As the solid amassed and coalesced toward the centerline of the flow, the point of liquid-solid phase transition propagated downstream of the initial channel throttle. Evidently,

alteration of flow geometry downstream of the initial throttle forced the flow's position of sonic transition downstream. As the plane of phase transition propagated downstream, solid argon previously formed upstream of the plane would dissipate. Meanwhile, solid accumulation proceeded down the channel, with the majority of adhered solid blowing out of the channel at irregular intervals. Solid accumulation immediately resumed at the channel throttle and proceeded downstream in a similar manner. In all cases in this pressure regime, the solid would not completely plug the channel. Either the solid would blow out repeatedly or a steady solid argon throttle would form near the exit plane of the channel. For long runs, the temperature of the channel walls cooled to the point where a solid argon throttle was able to stabilize at the channel exit, providing a steady artificial orifice, and partially restricting the exhaust flow.

With the reservoir providing saturated liquid argon at 40 psia, *moderate range*, formation of the solid proceeded similarly to that described for the *high range* (ref. Figure 3c). This was accompanied by the formation of solid along the interior of the channel orifice. This indicated that static pressures along portions of the orifice walls were beneath the triple point pressure, forcing transition to solid and accumulation on the orifice walls. The key difference between *high* and *moderate range* flows was that, as the argon delivery pressure was reduced, the location of phase transition moved from the wake of the throttled flow, upstream into the orifice itself. Solid transition was witnessed in the expansion wake of the orifice for the *high range*.

Solid accumulation inside the orifice exit was followed by a growth of solid upstream along the walls of the orifice. "Fingers" of solid were observed to grow into the plenum region along the walls, extending from the solid accumulated in the orifice. The "fingers" quickly dissipated and reformed in a random and transient manner. Although the static pressure in the plenum was measured to be well above the triple point, solidification in the plenum indicated a low pressure extension along the walls of the plenum. This appeared to be a phenomena developed due to the porosity of the solid accumulated in the orifice. As previously noted for the *high range*, the adhered solid blew out of the channel at irregular intervals and reformed at the throttle. Although solid accumulated in the orifice, the channel was never completely blocked in this pressure regime. Eventually, the channel walls cooled as the flow proceeded, and a solid argon throttle stabilized at the channel exhaust.

With the saturated liquid argon delivery reduced to 30 psia, *slight range*, the channel plugged almost immediately (ref. Figure 3d). Solid accumulated in the channel throttle, coalescing to form a solid plug. This formed a vapor lock in the manifold, positioning a layer

of liquid above the solid. Atop the liquid was gaseous argon, trapped by the plug on one end and the dewars on the other. The solid layer slowly grew into the liquid layer, propagating upstream. As the edge of the solid layer passed the plenum pressure tap, the pressure was noted to drop from 30 psia in the liquid to the vacuum chamber pressure in the solid. This indicated that the solid is sufficiently porous to allow good pumping of any gases trapped within the pores. This created a large pressure drop at the interface between the porous solid and the liquid. Solid formation continued until a "finger" from the solid layer reached the gaseous layer. At this point the plug would either partially or completely blow out of the channel. This relieved the vapor lock, providing liquid delivery to the channel. Immediately, plug formation resumed in a similar manner. Again the solid plug would blow out of the channel when the solid propagated through the liquid layer making contact with vapor locked gas. Occasionally, the plane of transition would migrate to the exhaust plane of the channel, as witnessed in the *high and moderate range*, where a cyclic process of plugging and blowout was established.

The channel never plugged permanently, but plug cycling time was directly related to the quantity of liquid trapped above the solid plug. Presumably, the plug would be permanent, neglecting sublimation and thermal loads from the channel walls, if the vapor locked gas was relieved, providing an unlimited quantity of liquid for solid transition. Solid growth rates were less than 1/2" per minute, in a 1" square cross-section.

Additional tests were conducted with saturated liquid nitrogen at delivery pressures from 35 psia to 17.5 psia. Due to the relatively low triple point pressure of nitrogen (1.82 psia), tests were limited by gas accumulation in the evacuated dump tank. Solid formation was similar to argon accumulation in the *high range* (ie. 65-50 psia). Namely, solid accumulated downstream of the channel throttle and migrated toward the channel exhaust, blowing out at irregular intervals. No solid accumulated in the channel orifice. The channel was never completely plugged.

For all liquid nitrogen flows the pressure ratio between delivery and triple point was greater than 9. Referencing the argon rating system, these flows would be attributed an *extremely high rating* where no accumulation in the channel was noted. But these flows produced solid accumulation similar to argon flows in the *high range*. Evidently, solid nitrogen was capable of accumulating downstream of the orifice due to the lack of momentum from the relatively low pressure nitrogen delivery. Therefore, pressure ratios for the *high range* of nitrogen must be enlarged. Regarding the *moderate and slight ranges*, some adjustment is expected due to the

reduced momentum and shear forces compared to argon flows in these ranges. The effect will be a contraction to the lower limit of the *moderate range*, providing an initiation of complete blockage at a slightly higher pressure. But the onset of the *moderate range* should be similar since formation of solid within the orifice is dependent on achieving pressures beneath the triple point within the orifice.

Discussion

Development of cyclical solid plugs and steady solid argon throttles poses some interesting questions about the structure of solid argon produced by adiabatic depressurization. Solid argon, which adheres to the wall and creates a throttling orifice, coexists with flowing gaseous and liquid argon at the surface of the solid. This may only occur for a pressure and temperature precisely at the triple point. Yet, pressure measurements in the flow upstream of the orifice indicate that pressures are greater than the triple point pressure. Therefore, all surfaces of solid argon, which provide an interface with the fluid phases at higher pressure, must reduce the fluid pressure and temperature at the interface to the triple point. Otherwise, heat input from the fluid would erode the solid. Therefore, a portion of the fluid at the interface must be pumped through the solid, reducing the interface pressure. This may only be explained if solid argon is formed as a porous medium during an adiabatic depressurization below the triple point.

The porous nature of the solid is especially evident when the flow channel plugs completely. The solid phase is observed to propagate upstream into the higher pressure liquid phase. Assuming the solid is porous, we propose the mechanism of solid growth and its evolution goes as follows. Gas within the porous solid, at pressures less than the triple point pressure, makes contact with liquid argon at the upstream interface of the propagating solid. Therefore, the surface of contact must be exactly at the triple point. Due to pumping through the porous solid, the pressure at the interface will tend to drop, causing liquid on the interface to transition into a mixture of solid and vapor phases, further forming additional porous solid. By this method, the solid propagates into the liquid phase. But growth would be stopped if gas, at pressures higher than the triple point, comes into contact with the porous solid. This occurs when the solid propagates through the entire liquid layer and makes contact with the overlaying layer of trapped gas. The gas then flows through the porous solid, producing a shear force at the interface between the solid argon and channel walls. Solid adherence to the walls has been weakened due to heat input from the

channel walls. Therefore, the shear force encounters little resistance, and the plug is blown out of the channel. In addition, gas contacting the porous solid can flow through the solid, raising the pressure and temperature. Under such conditions the solid can transition back into a fluid (liquid and gas), flowing out of the channel due to dewar pressure, providing an additional blowout mechanism.

In general, porous solids are formed when a fluid pressure falls through the triple point pressure. The heat of fusion must be released from the liquid in order to create the solid form. This heat is absorbed by the neighboring saturated liquid, where all heat input translates into vaporization. Therefore, the solid is formed as a porous medium, leaving gaps where the liquid has vaporized. The percentage of mass transforming into solid and gas phases is proportional to the ratio of heats of vaporization and fusion, where heat released to provide fusion is absorbed to produce vapor. Usually, the heat of vaporization is much greater than the heat of fusion. Therefore, more solid than gas is produced during transition. The porosity of the solid may be determined if the heat of vaporization, heat of fusion, and solid density are known for a given initial volume of liquid.

Returning to the question of hydrogen behavior in a throttled flow, nitrogen appears to provide the most analogous thermodynamic substitute, noting similarities of triple point pressure and ratios of heats for vaporization to fusion. Unfortunately, the low triple point pressure of nitrogen made the test durations fairly short. Moreover, providing nitrogen delivery pressures in the *moderate* and *slight ranges* would require partial evacuation of the cryogenic dewar, which is only rated for positive internal pressurization, in order to supply liquid at pressures less than atmospheric. But combining the results of the argon and nitrogen tests, one may estimate that a ratio of hydrogen delivery pressure to its triple point pressure of less than 5 ($P_s/P_{TP} < 5$, ~5 psia) will induce partial-to-complete blockage of the orifice. Greater hydrogen supply pressures should exhibit solid accumulation and adherence as demonstrated by nitrogen in the *high range*, where high pressure ratios produce varying degrees of accumulation and blockage in the flow channel downstream of the orifice. This accumulation is expected to reduce the flow rate by only a moderate amount. Alternatively, hydrogen supply pressures, with ratios to the triple point pressure of less than 5, should exhibit solid accumulation in the orifice and blockage similar to argon behavior in the *moderate* and *slight ranges*. This will substantially reduce and possibly block the flow.

Conclusions

Although future testing and analysis will help clarify and quantify the results, initial analysis, as described, appears fairly straightforward. The mechanism of solid formation as a cryogenic liquid depressurizes below its triple point is well displayed, providing insight into the phenomena.

From a practical standpoint, the experiment has provided a guideline to analyze potential hydrogen accumulation in the Centaur overboard exhaust vent. Namely, the degree of solid accumulation is a function of saturated liquid delivery pressure. Solid formation will not adhere to duct walls if the reservoir pressure and resulting purge flow rate is sufficiently large. Solid will accumulate in the duct below a definable pressure, but will partially, not completely, plug the duct if solid cannot precipitate inside the valve orifice.

For a hydrogen delivery pressure of 30 psia, as used by the Centaur, some solid hydrogen is expected to adhere to vent ducts. This adherence will not completely obstruct the vent flow, but will moderately reduce the flow rate if sufficient flow time is provided to generate a secondary solid hydrogen orifice within the vent. Partial plugs in the duct will moderately reduce the flow rate, due to the presence of two orifices. The coefficient of discharge from the duct will decrease due to the combination of the initial valve orifice in addition to the development of a hydrogen throttle. This additional throttle is expected to blowout occasionally while propagating toward the duct exit. This analysis is supported by an investigation conducted by Pratt & Whitney³, where adherence of solid hydrogen in the overboard vent duct was minimal for a supply pressure of 30 psia. Adherence of solid was observed to primarily be a function of delivery pressure, as well as heat input from the walls, momentum of the flow, flow time, and geometry.

Additional tests remain to be conducted on hydrogen to validate the results and subsequent analysis determined by tests conducted with argon and nitrogen flows. A reservoir pressure may be defined that will produce accumulation in the valve throttle, thereby providing the potential for complete plugging. Reservoir pressures must be maintained above this pressure to ensure cooling of the hydrogen turbopump during pre-ignition chill in the Centaur. Although not directly verified in this experiment, our data indicates that the 30 psia inlet pressure of the Centaur should be sufficient to prevent significant blockage of the overboard vent flow.

References

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† AIAA Member

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LIQUID PROPERTIES

Liquid	Hydrogen	Argon	Nitrogen	Carbon Dioxide
Triple Point Pressure	1.03	9.97	1.82	75.1
Triple Point Temperature	13.95	83.78	63.1	216.6
Triple Point Density (Liquid)	77	1415	868	1178
Triple Point Density (Vapor)	0.126	4	0.674	14
Ratio	611	354	1288	84
Heat of Vaporization	0.911	6.56	6.03	13.55
Heat of Fusion	0.117	1.19	0.724	8.65
Ratio	7.79	5.51	8.33	1.57
Surface Tension	2.31 @ 18K	13.2 @ 85K	10.53 @ 70K	9.13 @ 248K
Speed of Sound at Triple Point	281	169	161	220

Table 1

Argon Phase Diagram

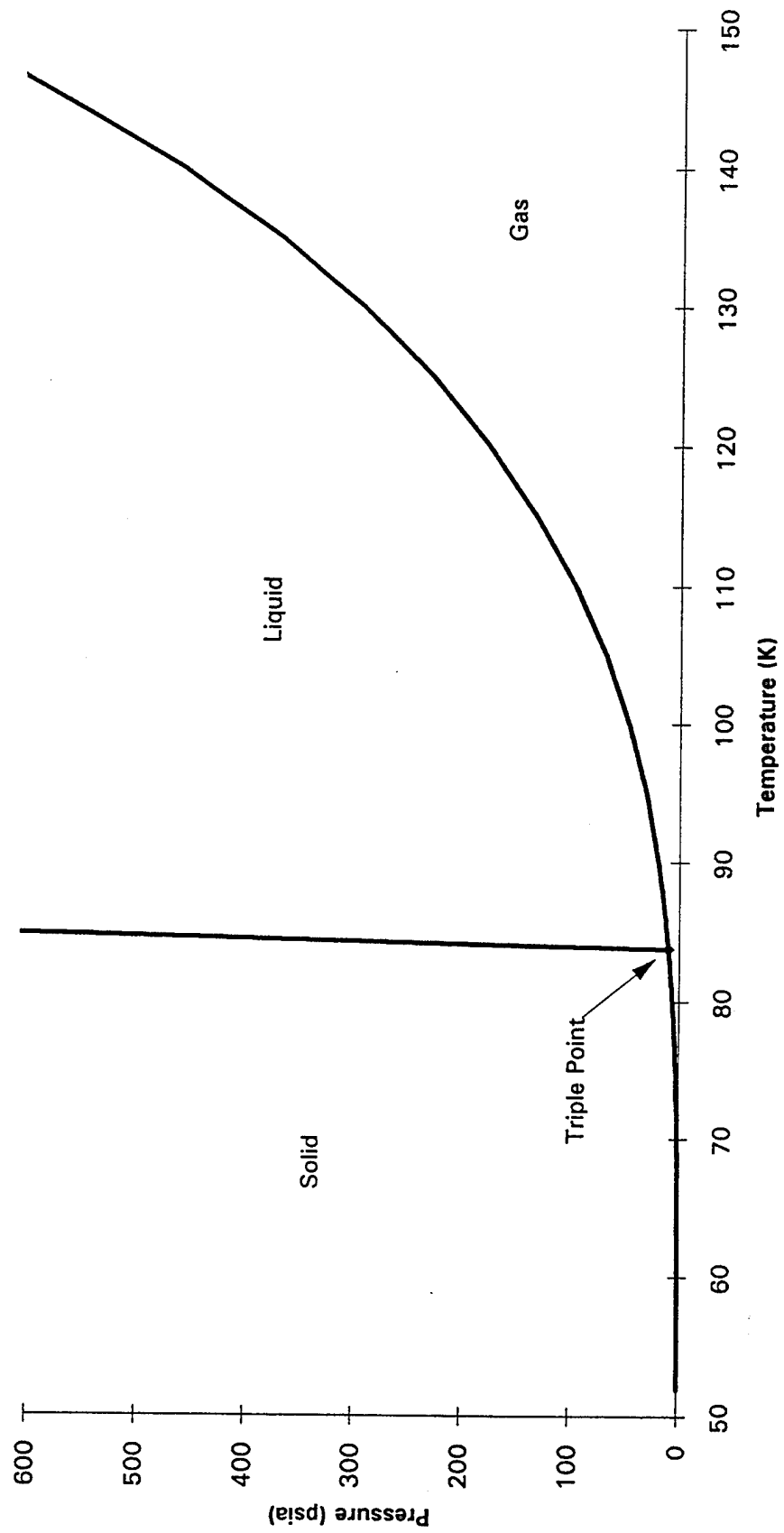


Figure 1

ARGON THROTTLE TEST SECTION

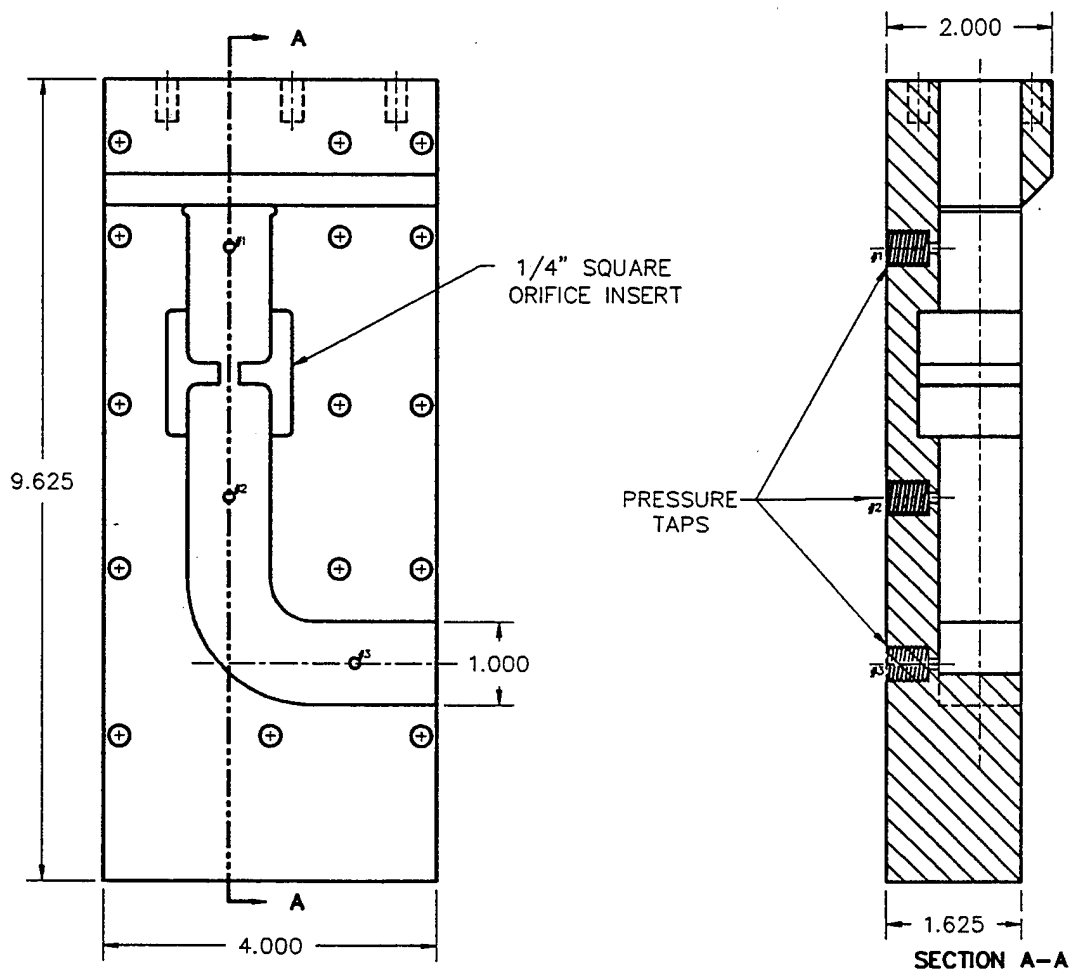


Figure 2

TYPICAL ACCUMULATION PHENOMENA AT FOUR DIFFERENT DELIVERY PRESSURES

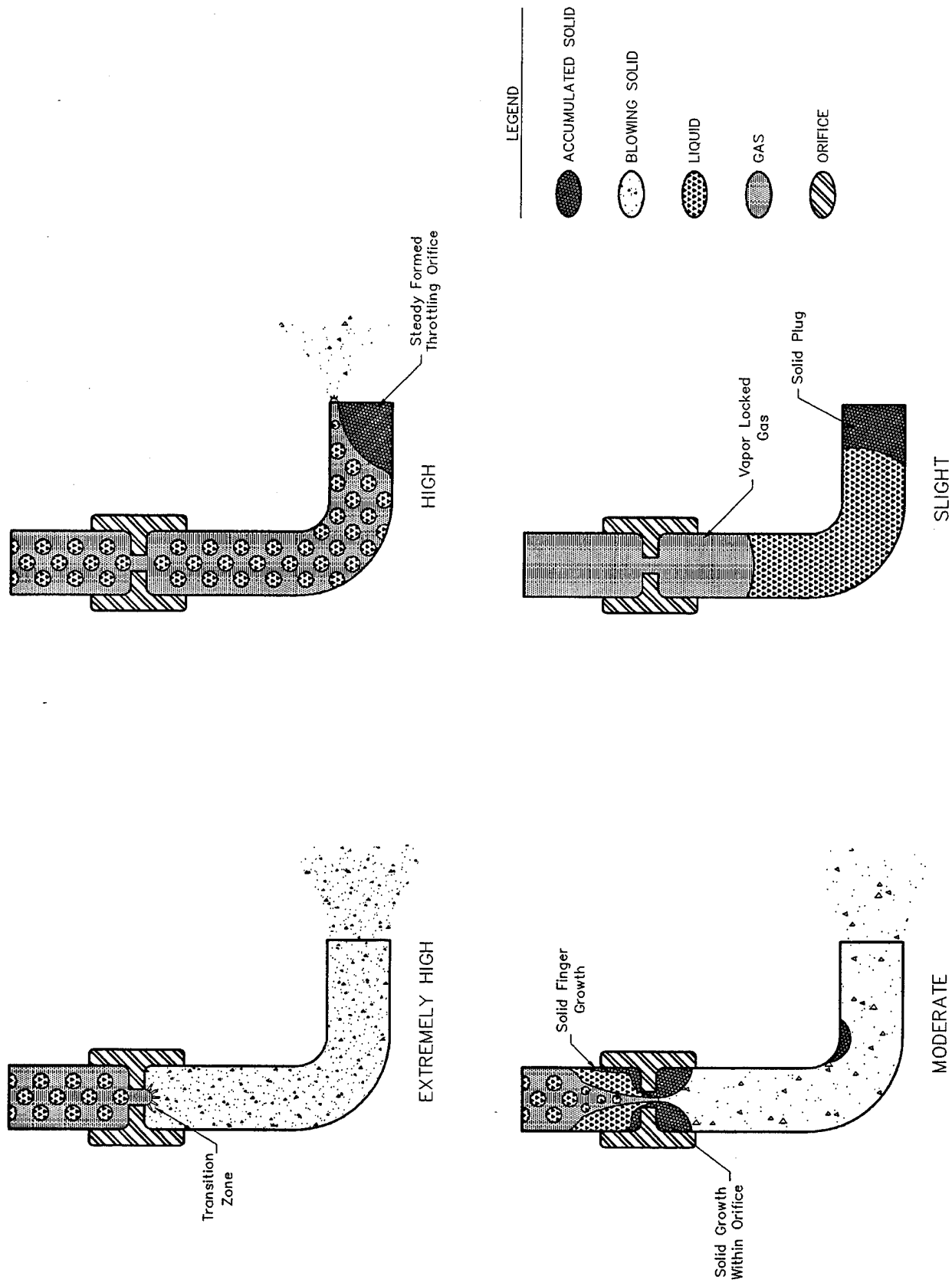


Figure 3

TECHNOLOGY OPERATIONS

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